

**SAMPLING PLAN FOR DISPERSION
OF FULLY-TREATED WATER**

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1. INTRODUCTION

On April 9, 2003, the U.S. Environmental Protection Agency Region 4 (EPA) issued permit no. OD 03-01 (Permit) to the Florida Department of Environmental Protection (FDEP or the Department) in accordance with the rules promulgated by EPA under the Marine Protection, Research, and Sanctuaries Act. The Permit authorizes the dispersion of fully-treated water in a 19,557 square mile area within the Gulf of Mexico provided 26 general and 27 specific conditions are met. The dispersion of treated water will begin as soon as a temporary pipeline is constructed to connect the abandoned Piney Point Phosphates, Inc. plant with Port Manatee in Manatee County, Florida. Figure 1 illustrates the regional location and Figure 2 illustrates the proposed pipeline route. Dispersion of treated water is expected to occur upon completion of the pipeline through November 30, 2003.

1.1 PURPOSE

Specific condition no. 22 of the Permit states:

The permittee shall submit for review and following approval by EPA, shall implement a wastewater quality verification plan prior to initiation of disposal activities. The purpose of the plan is to verify that the wastewater has not been contaminated as a result of contact with the pipeline, storage tanks, or vessel storage facilities. Based on the test results required as part of this plan, EPA will determine if the wastewater has been contaminated and whether and what permit modifications are appropriate. The plan shall include, but not be limited to:

- a. Identification of potential contaminants introduced by contact with the pipeline, storage tanks, or the vessel storage facilities;
- b. Collection procedures, frequencies and locations of wastewater samples to be collected at the Piney Point facility downstream of the acidulation station and from each disposal vessel; and
- c. The methods for analysis for the identified constituents.

This wastewater quality verification plan has been prepared to satisfy the requirements imposed by Specific Condition no. 22.

1.2 *FORMAT*

Section 2 describes the quality of the treated water to be transferred and the system that will be used to transfer the treated water from the final stage of the treatment process into the vessels docked at Port Manatee. Following these descriptions, Section 3 discusses the potential contaminants that could be introduced into the treated water by contact with the pipeline or vessel storage holds. Section 4 presents proposed sample collection procedures, locations, and frequencies as well as the proposed list of analytical constituents, the analytical methods, and the detection limits.

2. WATER TREATMENT AND TRANSFER SYSTEM

The water to be dispersed will be fully-treated utilizing an onsite lime precipitation, aeration, and sedimentation system. Following the final water quality treatment step, the water will be transferred to an unlined, abovegrade storage impoundment (called Basin No.2) on a continuous basis at a rate of approximately 1.0 to 3.0 million gallons per day (MGD). When a vessel chartered to transport water and disperse it in the approved area is docked at Port Manatee, a second, independent pipeline system will be used to transport the water from Basin No. 2 to the vessel. Each of these systems are briefly described in the following subsections.

2.1 ONSITE WASTEWATER TREATMENT SYSTEM

The Piney Point complex is equipped with a two-stage lime precipitation wastewater treatment circuit and an extended aeration/sedimentation pond that were constructed in 1980. The following paragraphs briefly describe the treatment process.

The first stage of the treatment circuit elevates the pH of the process wastewater from approximately 2.9 to 3.0 standard units to approximately 5.5 standard units using virgin slaked lime. This treatment step results in the precipitation of fluoride, metals, and radiological parameters.

The second stage of the lime treatment circuit processes first stage clarifier overflow (i.e. the clear water). In the second stage, the pH is elevated to above 11.5 standard units, again using virgin slaked lime. In the process, nearly all of the remaining fluoride and metals are precipitated as is phosphorus. Approximately 50 to 75 percent of the total dissolved solids are precipitated.

Importantly, elevation of the pH to above 11.5 standard units converts the dissolved ammoniacal nitrogen to the unionized form. As a result, spray aeration in the

downstream aeration pond cells reduces the total nitrogen content of the treated water to less than 40 milligrams per liter (mg/L) from over 600 mg/L. Reductions below 40 mg/L are possible when certain optimum conditions are experienced. The capacity of the system is estimated to be approximately 3.0 MGD.

Following aeration, the pH of the treated water is reduced by adding trace quantities of sulfuric acid in an automated acidulation station prior to offsite transfer. The acidulation station is capable of reducing the pH to within the 6.0 to 8.5 standard units range.

All of the water to be dispersed will be double-line treated (i.e., in both stages) and aerated. The total nitrogen content will vary dependent upon the flow rate through the aeration ponds as well as temporal conditions. Table 1 presents actual water quality analyses of water produced by this system in 2003. This data demonstrates that the quality of the water will consistently meet marine water quality standards, with the exception of ammonia, which sporadically will exceed the marine criteria for unionized ammonia. Copies of the certificates of analyses are available upon request.

2.2 BASIN NO. 2

Upon completion of the above-described water treatment process, the fully-treated water will be pumped from the acidulation station to Basin No. 2 in a high density polyethylene (HDPE) pipeline. Basin No. 2 is an existing seven-plus million gallon impoundment constructed principally above grade that historically was used as a sedimentation pond for non-contact non-process discharges of storm water runoff through former NPDES Outfall 001. Basin No. 2 is being retrofitted to serve as the treated water holding pond in lieu of use of tanks at either the Piney Point complex or Port Manatee. The retrofitting project consists of:

Table 1
2003 Analyses of Double Lime and Aeration Treated Water

Parameter	Units	No. of Samples	Averages	Marine Surface Water Standards
Acidity	mg/L	4	10.0	NS
Alkalinity -Total as CaCO ₃	mg/L	4	23.25	NS
Aluminum	mg/L	4	0.115	<1.5
Ammonia as N	mg/L	14	14.52	NS
Antimony	mg/L	4	0.022	<4,300
Arsenic	mg/L	4	0.007	<50
Barium	mg/L	4	0.005	NS
Beryllium	mg/L	4	0.004	<0.00013
BOD	mg/L	4	6.100	NS
Boron	mg/L	4	0.089	NS
Bromide	mg/L	4	0.250	NS
Cadmium	mg/L	4	0.004	<0.0093
Calcium	mg/L	4	650.0	NS
Chemical Oxygen Demand	mg/L	4	52.5	NS
Chloride	mg/L	4	98.0	+ 10%*
Chromium	mg/L	4	0.003	NS
Cobalt	mg/L	4	0.005	NS
Color	pcu	4	6.875	NS
Conductivity	µs/cm	4	20,888	NS
Copper	mg/L	4	0.005	<0.0037
Dissolved O ₂	mg/L	4	7.238	>5.0
Dissolved Orthophosphate	mg/L	4	0.083	NS
Fecal Coliform	/100mL	4	3.375	NS
Field pH	pH Units	4	6.903	+/- 1.0*
Field Temp	Deg. C	4	23.45	NS
Field Turbidity	mg/L	4	3.16	<29
Fluoride	mg/L	4	1.467	<1.5
Gross Alpha	pCi/L	4	13.1+/-5.4	<15
Gross Beta	pCi/L	4	144+/-10.7	NS
Iron	mg/L	4	0.025	<0.3
Lead	mg/L	4	0.003	<0.0085
Magnesium	mg/L	4	1.863	NS
Manganese	mg/L	4	0.002	<0.1
MBAS	mg/L	4	0.075	NS
Molybdenum	mg/L	4	0.025	NS
Nickel	mg/L	4	0.029	<0.0083
Nitrogen, Total as N	mg/L	14	16.742	NS
NO ₂ +NO ₃ as N	mg/L	14	0.806	NS
Oil/Grease	mg/L	14	4.3	5.0
Phenols	mg/L	14	0.016	1.0
Potassium	mg/L	14	133.81	NS
Radium 226	pCi/L	4	1.1+/-0.2	<5
Radium 228	pCi/L	4	0.5+/-0.3	<5

Parameter	Units	No. of Samples	Averages	Marine Surface Water Standards
Selenium	mg/L	4	0.015	<0.071
Silica	mg/L	4	4.950	NS
Silver	mg/L	4	0.011	NS
Sodium	mg/L	14	776	NS
Strontium	mg/L		1.650	NS
Sulfate	mg/L	14	3,495	NS
Sulfide	mg/L	4	0.500	NS
Sulfite	mg/L	4	1.550	NS
Thallium	mg/L	4	0.750	<6.3
Tin	mg/L	4	0.025	NS
Titanium	mg/L	4	0.024	NS
Total Chlorine	mg/L	4	0.050	NS
Total Dissolved Solids	mg/L	14	5,019	NS
Total Kjeldahl Nitrogen	mg/L	14	16.0	NS
Total Organic Carbon	mg/L	4	12.5	NS
Total Phosphorus as P	mg/L	4	0.340	NS
Total Suspended Solids	mg/L	14	6.990	NS
Uranium	pCi/L	4	3.0+/-1.1	NS
Vanadium	mg/L	4	0.005	NS
Zinc	mg/L	4	0.065	<0.086

Notes:

NS – No standard.

mg/L – Milligrams per liter.

pCi/L = Picocuries per liter.

µs/cm = Microhoms per centimeter.

* = When compared to background concentrations.

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- Dewatering the basin;
 - Excavating the base to clean soil;
 - Leveling (i.e., filling low spots) the dike crest elevation;
 - Installing discharge (from the acidulation circuit pipeline) diffuser baffles to minimize the potential to create turbidity; and
 - Installing a structure to hold the permanent pump and intake and the suction line of the standby pump for the pipeline to Port Manatee described below.

2.3 PORT MANATEE PIPELINE DESIGN

The 11,000-ft long transfer pipeline from the site to Port Manatee entails the construction of a 20-inch diameter steel line from the transfer pump station located within Basin No. 2 on the Piney Point property to a point 2,000 ft north of the transfer station where a connection to a 16-inch diameter, high density, heat-infused polyethylene line will be made (see Figure 2). The high-pressure steel pipeline will consist of bolted sections of flanged-end pipe laid on grade. The thick-walled pressure rated (SDR-11) polyethylene pipeline will be laid on grade except where roadway and ditch crossings are anticipated. One paved roadway crossing at the entrance road to the plant site and other smaller, shallow soil-covered crossings are contemplated. The 16-inch diameter heat-fused polyethylene line will cross beneath the Florida Department of Transportation (FDOT) right-of-way of US Highway 41 and its east and west drainage ditches at a point approximately 3,300 ft north of the transfer pump station. A right-of-way utilization permit and permission to pass the polyethylene line through a 4-ft by 8-ft concrete box drainage culvert has been acquired from FDOT. The pipe will enter the box culvert at its centerline and lay along its invert. After passing through approximately 140 linear feet of culvert, the pipeline will follow the ditch bank, cross the ditch invert, and enter into a 38-inch diameter, 78-ft long reinforced concrete pipe culvert and follow its invert. After crossing along the invert of the culvert, the pipeline will then be placed under an industrial spur track operated by Port Manatee. The crossing will involve a shallow trench cut under the track and burial inside a heavy wall, steel pipe casing. After passing through the steel casing, the pipeline will rise up and be placed on grade along the Port

Manatee right-of-way and then follow along the south right-of-way of Harlee Road to a point where a buried crossing is needed under the pavement of Harlee Road. After crossing under Harlee Road, the pipeline will rise up, then cross under a dirt road, and then follow the edge of a paved parking lot to a point where a sub-road crossing is intended at the intersection of Reeder Road and Piney Point road. This sub-road crossing will be through an existing concrete culvert. Upon exiting the concrete culvert the pipeline will lie along the west right-of-way of Reeder Road and Piney Point Road. This sub-road crossing will be through an existing concrete culvert. Upon exiting the concrete culvert the pipeline will lie along the west right-of-way of Reeder Road, which is maintained by Manatee County, then turn westerly, and follow along the north edge of the privately owned north Dock Street. The polyethylene pipeline will finally be connected to an existing steel manifold that serves a series of underground high-pressure steel lines owned and formerly used by Coastal Petroleum (Now Trans Montaigne) that run to berth-side pits at the Port Manatee dock. At these berth-side pits, two, eight inch, new rubber hoses that will be coupled to the steel lines and the vessel. These hoses will be disconnected between each vessel loading event and stored at Port Manatee.

3. POTENTIAL FOR CONTAMINANTS TO BE INTRODUCED DURING TRANSFER

The description of the transfer system in Section 2 provides the information necessary to address subpart a of Specific Condition no. 22 in the Permit. Based upon this description, the potential for contamination of the fully-treated water to occur during transfer to the dispersion site is limited to: (1) suspended solids and turbidity from discharge into and withdrawal from the unlined Basin No. 2 temporary storage pond; (2) (remnant jet fuel in the steel pipes at Port Manatee; and (3) remnant petroleum hydrocarbons in the cargo holds of the vessel(s). Each of these potential sources is discussed below.

3.1 BASIN NO. 2

Transfer of water into Basin No. 2 at a two to three MGD rate on a continuous basis, together with periodic withdrawals at a rate of four MGD, could increase the levels of suspended solids and turbidity in the fully-treated water. Also, because Basin No. 2 is open, there is the potential for the introduction of foreign matter such as tree leaves, twigs, and algae growth as well. There is, however, little probability of chemical contamination because the operating level of Basin No. 2 always will be above the groundwater table and because groundwater beneath this portion of the site is relatively unimpacted.

3.2 PETROLEUM CONTAMINATION FROM PORT PIPELINES

The steel pipelines owned by Trans Montaigne buried beneath Port Manatee most recently were used to transport jet fuel from berths 8, 9, and 10 to a nearby tank farm. These pipelines have been disconnected from the tank farm to prevent the possibility of petroleum contamination of the fully-treated water by operator error (i.e., improperly opening a valve). Thus, for the duration of the permit, the steel pipelines beneath Port Manatee, as well as all of the other pipelines used to transfer the fully-treated water, will be dedicated to these water transfers.

3.2.1 Pipeline Cleaning Procedures

However, there is a reasonable likelihood that residual petroleum could contaminate the fully-treated water, particularly during the initial transfers. Therefore, the pipeline will be cleaned using the following sequence of steps following the completion of construction and prior to its first use:

1. Push/pull absorbent pigs through the pipeline to remove free product, sludge, and scale;
2. Wash with N-SPEC-120[®] brand solvent; and
3. Rinse with clean water.

Steps (2) and (3) will be repeated until the water in the pipeline meets the requirements specified in Section 4.1.2. Product literature and the Material Safety Data Sheet (MSDS) for N-SPEC-120[®] are enclosed in Appendix A.

3.2.2 Minimum Pipeline Cleanliness

In order to conservatively protect marine water quality in the dispersion area, the pipeline will be cleaned such that the initial barge loads of water contain less than the following levels of pollutants that are indicative of the presence of petroleum hydrocarbons in general, and specifically jet fuel:

- Oil and grease < 5 milligrams per liter (mg/L);
- Detergents < 0.5 mg/L;
- Total residual petroleum hydrocarbons (TRPHs) < 5 mg/L;
- Lead < 5.6 µg/L; and
- Napthalene < 100 µg/L.

These values are the marine water quality standards developed by EPA except for naphthalene, for which there is no water quality standard. The naphthalene standard is the water quality based effluent standard applied by EPA and the Department at petroleum remediation sites that surface discharge water into marine environments.

Because the pipeline will be used solely to transport fully-treated water into vessels during the authorized dispersion period, there is no probability of recontamination once the pipeline is cleaned. All subsequent shipments should be cleaner than the initial shipment.

3.3 PETROLEUM CONTAMINATION FROM VESSEL(S)

The vessel(s) being considered for transporting and dispersing the fully-treated water have been used to move a variety of liquid petroleum products. For the duration of the approved dispersion, however, the vessel(s) will be dedicated to the transport and dispersion of water, thereby eliminating the potential for contamination of the water with residual petroleum hydrocarbons, provided the vessel(s) is (are) thoroughly cleaned prior to the initial shipment.

3.3.1 Vessel Cleaning Procedure

The following steps will be implemented to ensure the vessel(s) is (are) clean prior to loading the initial shipment:

1. Using vacuum trucks/diaphragm pumps, remove the remaining product from the tank bottoms;
2. Insert horn blowers into the butter-worth openings, air blow cargo tanks to remove any fumes, attempt to lower LEL readings for entry into cargo tanks;
3. Water wash internal tanks for removal of any previous product that could be present;
4. Using airless sprayer apply seawash detergent to internal walls and floor (MSDS on this product is contained in Appendix A);
5. Water wash walls and floor to remove detergent and any previous product from internals;
6. Using vacuum truck/diaphragm pumps, remove wash water and detergent from internal tanks for proper disposal;
7. Using ladders, remove any and all loose rust scale that could be settled on internal beams and walls for proper disposal; and

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8. Detergent wash piping and flush with water to remove previous product.

3.3.2 Minimum Vessel Cleanliness

In order to conservatively protect marine water quality in the dispersion area, the vessel will be cleaned such that the initial barge loads of water contain less than the following levels of pollutants that are indicative of the presence of petroleum hydrocarbons in general, and specifically jet fuel:

- Oil and grease < 5 milligrams per liter (mg/L);
- Detergents < 0.5 mg/L;
- TRPHs < 5 mg/L;
- Lead < 5.6 µg/L; and
- Naphthalene < 100 µg/L.

These values are the marine water quality standards developed by EPA except for naphthalene, for which there is no water quality standard. The naphthalene standard is the water quality based effluent standard applied by EPA and the Department at petroleum remediation sites that surface discharge water into marine environments.

Because the vessel will be used solely to transport fully-treated water into vessels during the authorized dispersion period, there is no probability of recontamination once the vessel is cleaned. All subsequent shipments should be cleaner than the initial shipment.

4. SAMPLING AND ANALYSIS PLAN

Consideration of the information presented in Sections 2 and 3 above results in a logical confirmation sampling protocol that is divided into two phases: (1) prior to initial shipments; and (2) initial shipment. The objective to confirm that the pipeline and vessel cleaning procedures have been effective and that contact with these surfaces will not cause the water to exceed the cleanliness criteria presented in Section 3.3.2 and 3.4.2. Of course, the following sampling protocol is based upon the current plans to use (a) dedicated vessel(s) to transport and disperse the water and to physically preclude use of the Trans Montaigne pipelines for other purposes throughout the dispersion period. If these plans change, the sampling and analysis protocol may need to be revisited.

4.1 PHASE I PRIOR TO INITIAL SHIPMENTS

Prior to transferring any water from the Piney Point complex, it will be necessary to confirm that the procedures for cleaning the pipeline and the vessel(s) have been effective. Accordingly, a series of interim samples will be collected to provide guidance to the cleaning contractors concerning the need to conduct additional cleaning and rinsing cycles. Once the cleaning contractors complete the final washing step, the final rinse water will be sampled and analyzed to confirm that the initial shipment will meet the specifications listed in Section 3.3.2 and 3.4.2. The following sampling and analysis protocols will apply to both the pipeline and the vessel(s) cleanings.

4.1.1 Interim Sampling and Analyses

As the pipeline and vessel cleaning contractors progress through the cleaning and rinsing steps described in Section 3.3.1 and 3.4.1 above, they will contact the site Receiver's consultants (Ardaman & Associates and Environmental Consulting & Technology, Inc. [ECT]) when they believe the pipeline or vessel is clean. ECT technicians will then visit the cleaning site and collect grab samples of the rinse water. In order to provide

immediate guidance to the cleaning contractors, ECT will utilize the following field analytical techniques:

- Turbidity: calibrated meter;
- pH: calibrated meter;
- TRPH: colorimetric kit; and
- Lead: pocket colorimeter II[®].

Appendix B contains information about these analytical methods. If analyses using these methods indicate that the levels of TRPH and lead in the rinse water are less than 10 times the cleanliness levels listed in Section 3, the final confirmation samples will be collected as described below. If these field analyses indicate that the rinse water contains more than 10 times the cleanliness levels listed in Section 3, the cleaning contractors will be advised to repeat their wash and rinse cycles and advise ECT when to return to repeat these procedures. (Note: the application of 10 times the cleanliness criteria is based upon the fact that not more than 100,000 gallons of rinse or hydrostatic test water will be produced and that the minimum barge size tendered has a one million gallons capacity. Thus, a minimum dilution of 10 times will occur with the first shipment. As a result, if the final rinse water contains less than 10 times the prescribed cleanliness levels, the initial shipment will meet these water quality based standards.)

4.1.2 Final Sampling and Analysis

Once the pipeline and vessel(s) cleaning rinse waters meet the target levels of TRPH and lead, final samples will be collected and submitted to the Severn Trent Laboratory in Tampa for analyses using the methods listed in Table 2 below.

Table 2
Verification Sampling Analytes

Analyte	Method	Detection Limit
Oil and Grease	EPA 1664	0.89 mg/L
Surfactants (MBAs)	EPA 425	0.039 mg/L
TRPH	FL-PRO	0.1 ppm
Lead	EPA 200.7	1.5 ppb
Naphthalene	EPA 625	0.36 ppb

These will be grab samples collected in accordance to FDEP's standard quality assurance operating procedures prescribed by Chapter 62-160 of the Florida Administrative Code. Samples will be placed in laboratory supplied sample containers, chilled in ice to less than 4 degrees Celsius (4° C), and transported to the laboratory immediately after the sampling event has been completed. The laboratory will be instructed to report the results within the shortest period possible after receipt of the samples. If the laboratory analyses indicate that the levels of potential contaminants are less than 10 times the specified cleanliness levels, then the pipeline and/or barge will be declared ready to accept shipments of fully-treated water.

4.2 PHASE II INITIAL SHIPMENT

As the initial shipment is being loaded, a series of grab samples will be collected from the cargo holds of and analyzed using the field test sampling procedures and methods described in Section 4.1.1. These results will be used to indicate whether any apparent contamination is being detected in excess of the cleanliness levels prescribed in Section 3.3.2.

In addition, 5 discrete samples will be collected immediately downstream from both the acidulation station and from the cargo hold as the ship is being loaded. Each cargo hold discrete sample will be a manual composite sample collected from between 3 to 5 randomly selected areas or compartments within the cargo hold. The samples will then be transferred into laboratory-supplied containers, placed in ice to chill to less than 4° C, and transported to Severn Trent's Tampa laboratory immediately after loading of the vessel is completed. Laboratory analyses of the composite sample will be conducted for the parameters listed in Section 4.1.2 using those same analytical methods. The laboratory will be instructed to report the results in the shortest possible time period.

The results will be forward to EPA in order to determine whether that the wastewater has not been contaminated. In addition, the results will be used to determine the degree of accuracy of the field analytical techniques.

4.3 ONGOING CONFIRMATION SAMPLING AND ANALYSIS

As noted earlier in this plan, the probability of contamination of the wastewater through contact with the pipeline or vessel will be the greatest during the initial shipment due to the prior use of this equipment for the transportation of petroleum products. Use of dedicated equipment lessens the probability of contamination with each subsequent shipment. Therefore, sampling and analysis of subsequent shipments will be limited to testing the turbidity of the water after it is withdrawn from Basin No. 2. A calibrated meter will be used to measure the quality of the water being transferred.

FIGURES

APPENDIX A

MATERIAL SAFETY DATA SHEET N-SPEC120 CLEANER

Material Safety Data Sheet

Section 1. Chemical Product and Company Identification

Common Name	N-SPEC 120 Cleaner	Code	Not available.
Supplier	Coastal Fluid Technologies A Division of Coastal Chemical Co., L.L.C. 3520 Veterans Memorial Drive Abbeville, LA 70511 318-893-1952	MSDS#	Not available.
Synonym	Not available.	Validation Date	11/29/1999
Trade name	Not available.	Print Date	02/14/2000
Material Uses	Not available.	In case of Emergency	TRANSPORTATION EMERGENCY: Chemtec 1-800-424-9300
Manufacturer	Coastal Fluid Technologies A Division of Coastal Chemical Co., L.L.C. 3520 Veterans Memorial Drive Abbeville, LA 70511 318-893-1952		

Section 2. Composition and Information on Ingredients

Name	CAS #	% by Weight	TLV/PEL	LC ₅₀ /LD ₅₀
1) Confidential Infomation				

Section 3. Hazards Identification

Emergency Overview	<p>WARNING!</p> <p>MAY BE HARMFUL IF SWALLOWED.</p> <p>CONTAINS MATERIAL WHICH CAN CAUSE SPECIFIC ORGAN OR SYSTEM DAMAGE: (blood, the nervous system, upper respiratory tract, eyes).</p> <p>Causes severe eye irritation.</p> <p>MAY CAUSE SKIN IRRITATION.</p>
Routes of Entry	Eye contact. Inhalation. Ingestion.
Potential Acute Health Effects	Hazardous in case of eye contact (irritant), of ingestion, of inhalation. Hazardous in case of skin contact (irritant). Slightly hazardous in case of skin contact (sensitizer). Severe over-exposure can result in death. Inflammation of the eye is characterized by redness, watering, and itching.
Potential Chronic Health Effects	<p>CARCINOGENIC EFFECTS: Not available.</p> <p>MUTAGENIC EFFECTS: Not available.</p> <p>TERATOGENIC EFFECTS: Not available.</p> <p>DEVELOPMENTAL TOXICITY: Not available.</p> <p>The substance is toxic to the nervous system, upper respiratory tract, eyes, blood.</p> <p>Repeated or prolonged exposure to the substance can produce target organs damage. Repeated exposure to an highly toxic material may produce general deterioration of health by an accumulation in one or many human organs.</p>

Section 4. First Aid Measures

Eye Contact	Check for and remove any contact lenses. IMMEDIATELY flush eyes with running water for at least 15 minutes, keeping eyelids open. COLD water may be used. DO NOT use an eye ointment. Seek medical attention.
Skin Contact	After contact with skin, wash immediately with plenty of water. Gently and thoroughly wash the contaminated skin with running water and non-abrasive soap. Be particularly careful to clean folds, crevices, creases and groin. COLD water may be used. Cover the irritated skin with an emollient. If irritation persists, seek medical attention. Wash contaminated clothing before reusing.
Hazardous Skin Contact	Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek immediate medical attention.

Continued on Next Page

Inhalation	Allow the victim to rest in a well ventilated area. Seek immediate medical attention.
Hazardous Inhalation	No additional information.
Ingestion	DO NOT induce vomiting. Examine the lips and mouth to ascertain whether the tissues are damaged, a possible indication that the toxic material was ingested; the absence of such signs, however, is not conclusive. Loosen tight clothing such as a collar, tie, belt or waistband. If the victim is not breathing, perform mouth-to-mouth resuscitation. Seek immediate medical attention.
Hazardous Ingestion	No additional information.

Section 5. Fire and Explosion Data

Flammability of the Product	Not available
Auto-Ignition Temperature	Not available.
Flash Points	CLOSED CUP: >93.333°C (200°F).
Flammable Limits	Not available.
Products of Combustion	These products are carbon oxides (CO, CO ₂), sulfur oxides (SO ₂ , SO ₃ ...).
Fire Hazards in Presence of Various Substances	Not available.
Explosion Hazards in Presence of Various Substances	Risks of explosion of the product in presence of mechanical impact: Not available. Risks of explosion of the product in presence of static discharge: Not available. Slightly explosive to explosive in presence of oxidizing materials.
Fire Fighting Media and Instructions	Flammable liquid, soluble or dispersed in water. SMALL FIRE: Use DRY chemicals, CO ₂ , alcohol foam or water spray. LARGE FIRE: Use alcohol foam, water spray or fog.
Special Remarks on Fire Hazards	No additional remark.
Special Remarks on Explosion Hazards	No additional remark.

Section 6. Accidental Release Measures

Small Spill	The concentrated form of this material is a cleaner. During application, hazardous material on the apparatus or structure being cleaned may become part of the cleaning solution. Check with all applicable regulations before disposing of the material created during application.
Large Spill	The concentrated form of this material is a cleaner. During application, hazardous material on the apparatus or structure being cleaned may become part of the cleaning solution. Check with all applicable regulations before disposing of the material created during application.

Section 7. Handling and Storage

Handling	Not available.
Storage	Keep container tightly closed and in a well-ventilated place.

Section 8. Exposure Controls/Personal Protection

Engineering Controls	Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the work-station location.	
Personal Protection	Safety glasses. Lab coat. Gloves (impervious).	
Personal Protection in Case of a Large Spill	Splash goggles. Full suit. Boots. Gloves. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.	
Chemical Name or Product Name	CAS #	Exposure Limits
1) Confidential Information		

Section 9. Physical and Chemical Properties

Physical state and appearance	Liquid.	Odor	Not available.
Molecular Weight	Not applicable.	Taste	Not available.
pH (1% soln/water)	6 to 8 [Neutral.]	Color	Blue. (Dark.)
Boiling Point	The lowest known value is 100°C (212°F) (Water). Weighted average: 140.43°C (284.8°F)		
Melting Point/Pour Point oC	-8.8889°C (16°F)		
Critical Temperature	Not available.		
Specific Gravity	0.96 to 0.98 (Water = 1)		
Vapor Pressure	The highest known value is 17.2 mm of Hg (@ 20°C) (Water). Weighted average: 8.77 mm of Hg (@ 20°C)		
Vapor Density	The highest known value is 5.11 (Air = 1). Weighted average: 2.93 (Air = 1)		
Volatility	Not available.		
Odor Threshold	The highest known value is 34.6 ppm		
Evaporation rate	0.02 compared to Butyl acetate		
Viscosity	Not available.		
Water/Oil Dist. Coeff.	The product is much more soluble in water.		
Ionicity (in Water)	Anionic.		
Dispersion Properties	See solubility in water, methanol, diethyl ether.		
Solubility	Easily soluble in cold water, hot water, methanol, diethyl ether. Insoluble in n-octanol.		
Physical Chemical Comments	Not available.		

Section 10. Stability and Reactivity Data

Chemical Stability	The product is stable.
Conditions of Instability	No additional remark.
Incompatibility with various substances	Reactive with oxidizing agents, acids. Slightly reactive to reactive with reducing agents.
Hazardous Decomposition Products	Not available.
Hazardous Polymerization	No.

Section 11. Toxicological Information

Toxicity to Animals	Acute oral toxicity (LD50): 1900 mg/kg [Rat]. Acute dermal toxicity (LD50): 9510 mg/kg [Rabbit].
Chronic Effects on Humans	No additional remark.
Other Toxic Effects on Humans	Hazardous in case of eye contact (irritant), of ingestion, of inhalation. Hazardous in case of skin contact (irritant). Slightly hazardous in case of skin contact (sensitizer).
Special Remarks on Toxicity to Animals	No additional remark.
Special Remarks on Chronic Effects on Humans	No additional remark.
Special Remarks on other Toxic Effects on Humans	Material is irritating to mucous membranes and upper respiratory tract.

Section 12. Ecological Information

Ecotoxicity	Not available.
BOD5 and COD	Not available.
Products of Biodegradation	Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.
Toxicity of the Products of Biodegradation	The products of degradation are less toxic than the product itself.
Special Remarks on the Products of Biodegradation	No additional remark.

Section 13. Disposal Considerations

Waste Disposal	Recycle, if possible. Consult your local or regional authorities.
----------------	---

Section 14. Transport Information

Propper Shipping Name	NOT REGULATED
DOT Classification	Not a DOT controlled material (United States).
DOT Identification Number	NONE
Packing Group	Not applicable.
Hazardous Substances Reportable Quantity (kg)	11061.8lbs. (5016.7 kg)
Special Provisions for Transport	Contains alkylbenzenesulfonate

Section 15. Regulatory Information

Federal and State Regulations TSCA inventory: All components

Other Classifications	WHMIS (Canada)	WHMIS CLASS D-2A: Material causing other toxic effects (VERY TOXIC).
	DSCL (EEC)	R11- Highly flammable.

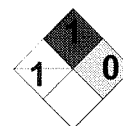
Section 16. Other Information

HMIS (U.S.A.)

Health Hazard	*	1
Fire Hazard		1
Reactivity		0
Personal Protection		B

National Fire Protection Association (U.S.A.)

Health



Fire Hazard

Reactivity

Specific hazard

References Not available.

Other Special Considerations No additional remark.

Validated by Joe Hudman on 11/29/1999.

Verified by Joe Hudman.

Printed 02/14/2000.

Emergency Contact Chemtrec 1-800-424-9300

Notice to Reader

To the best of our knowledge, the information contained herein is accurate. However, neither the above named supplier nor any of its subsidiaries assumes any liability whatsoever for the accuracy or completeness of the information contained herein. Final determination of suitability of any material is the sole responsibility of the user. All materials may present unknown hazards and should be used with caution. Although certain hazards are described herein, we cannot guarantee that these are the only hazards that exist.

SEAWASH**MATERIAL SAFETY DATA SHEET**

This Material Safety Data Sheet is primarily directed to managerial, safety and medical persons. The information contained within meets the Material Safety Data Sheet (MSDS) requirements of the federal OSHA Hazard Communication Standard (29 CFR 1910.1200).

SECTION ONE: IDENTIFICATION

Product Trade Name: SEAWASH	Manufacturer: Warren Chemical Corp.
Description: Concentrated All-Purpose Cleaner	P.O. Box 545 (Hwy 190)
Chemical Formula: Proprietary	Robert, LA 70455
Chemical Family: Surfactants (mixture)	Emergency Telephone: 504-542-8985

SECTION TWO: HAZARDOUS INGREDIENTS

Hazardous Components: None. SEAWASH contains no materials listed as hazardous (as defined in the OSHA Hazard Communication Standard).

SECTION THREE: PHYSICAL DATA

Appearance and Odor: SEAWASH is a clear and free-flowing liquid, with a slight rose odor.	
Boiling Point: 210 F	Specific Gravity: 1.083
Solubility in Water: Infinite	pH: 11.0

SECTION FOUR: FIRE AND EXPLOSION HAZARD DATA

SEAWASH is non-flammable and will not support combustion.

Flashpoint: None

Method Used: Open cup

Special Fire Fighting Procedures: Not applicable to SEAWASH itself. Use standard procedures for extinguishing surrounding materials.

SECTION FIVE: HEALTH HAZARD DATA

Health Hazards: None. SEAWASH is non-toxic. **Effects of Overexposure:** Not Applicable.

Emergency First Aid Procedures: If concentrate gets into eyes, flush thoroughly with water.

continued on back

APPENDIX B

PROPOSED FIELD ANALYTICAL METHODS

Hach Scanning Analyzer

HSA-1000

For Lead and Copper

The portable HSA-1000 is an easy-to-use, accurate, cost-effective instrument for monitoring lead and copper concentrations. Using differential pulse anodic stripping voltammetry, a USEPA-approved methodology for determining total lead in drinking water when used with acid digestion*, the HSA-1000 makes monitoring easy.

Simply dissolve a conditioning tablet in your sample and immerse the unique, disposable, patented electrode to measure the parameter you need. The HSA-1000 instrument does the rest, giving you parts-per-billion results in about three minutes.

And with the convenience of the HSA-1000 (it's battery-operated and comes with a soft-sided carrying case) you can easily take the instrument, manual and sensors to any point in your distribution system for on-site testing.

Once you get your results, managing them is just as easy. You can store your data and recall it to the instrument display for later analysis, or download it to a computer system or printer using the RS-232 serial interface.

The HSA-1000 is easy-to-use, portable, and affordable. It costs much less than outsourcing your testing, and less than purchasing an alternative testing instrument such as an atomic absorption unit. Plus, you receive the technical support and customer service from Hach you know you can count on.

The Hach Scanning Analyzer HSA-1000. It's another example of high technology made simple in a test you can trust, from Hach.

*The HSA-1000 method is approved by the USEPA only for determining lead concentration, and acid digestion is required for reporting purposes.



www.hach.com

To order, call 800.227.4224 or e-mail orders@hach.com.

For a quote, fill out the quotation form on our web site at www.hach.com and we'll get you the information you need, usually within 24 hours.

Hach Scanning Analyzer HSA-1000

System Performance Characteristics

Parameter	Lead	Copper
Calibration	Pre-calibrated electrodes (HSE-Pb)	Pre-calibrated electrodes (HSE-Cu)
Analysis Time	3 minutes	3 minutes
Range	2-100 µg/L	50-2000 µg/L
Detection Limit	2 µg/L	50 µg/L
Resolution	1 µg/L	1 µg/L
Precision	+/- 5% CV at 15 µg/L	+/- 5% CV at 250 µg/L
Operating Temperature	15-30°C	15-30°C



Specifications:

Internal memory: Stores up to 300 readings
 Display: Intelligent 2 x 16 character alphanumeric
 Output interface: Output to printer or computer via RS-232 serial interface
 Power: Uses eight AA 1.5v batteries, auto switch-off
 Size: Instrument only, 170 x 130 x 55 mm

Ordering Information:

*The HSA-1000 instrument tests for both lead and copper.
 Just order the replacement sensor pack for the parameter you need.*

50400-00 HSA-1000 Analyzer, plus Sensor Pack for Lead
 Includes instrument, manual, soft-sided carrying case, one sensor pack for lead, two electrode caps and test tube holders, eight AA batteries

50400-10 HSA-1000 Analyzer, plus Sensor Pack for Copper
 Includes instrument, manual, soft-sided carrying case, one sensor pack for copper, two electrode caps and test tube holders, eight AA batteries

50401-00 Sensor Pack for Lead (10 tests)
 Includes 10 HSE-Pb scanning electrodes, 10 PrepTab™ PT-Pb sample preparation tablets, 10 sample tubes, crushing rods

50402-00 Sensor Pack for Copper (10 tests)
 Includes 10 HSE-Cu scanning electrodes, 10 PrepTab™ PT-Cu sample preparation tablets, 10 sample tubes, crushing rods



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All other inquiries, contact:

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Product Information

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Pocket Colorimeter II Analysis System: Product Overview

The new Pocket Colorimeter™ II Filter Photometer is a true go-anywhere instrument, lightweight and battery operated, suitable for extended field work or quick, on-the-spot process monitoring.



- **Waterproof, Lightweight, Economical**

The Pocket Colorimeter II Instrument really can be carried in your pocket! Weighing in at only .23 kg (8.1 oz), it even floats if accidentally dropped in water.

- **Accurate, Reproducible Measurements**

Despite accuracy comparable to a lab instrument, the low-cost Pocket Colorimeter II is simple enough to use that anyone can obtain reliable results.

- **Factory-Programmed**

Each Pocket Colorimeter II Instrument is pre-programmed for one of more than 35 parameters; many are based on EPA-approved methods. No manual calibration is ever required. Simply zero the instrument with a blank, insert the reacted sample, and read the result.

- **Improved Optics**

A long-lasting LED light source allows expanded ranges for ammonia, chlorine, chromium, copper, iron, and molybdenum, reducing the need for dilutions.

The Pocket Colorimeter II allows user calibrations, giving you the ability to create your own calibration curve, or to perform a standard adjust. We also manufacture wavelength-specific 'generic' instruments, allowing you to enter your own methods using from two up to ten standards.

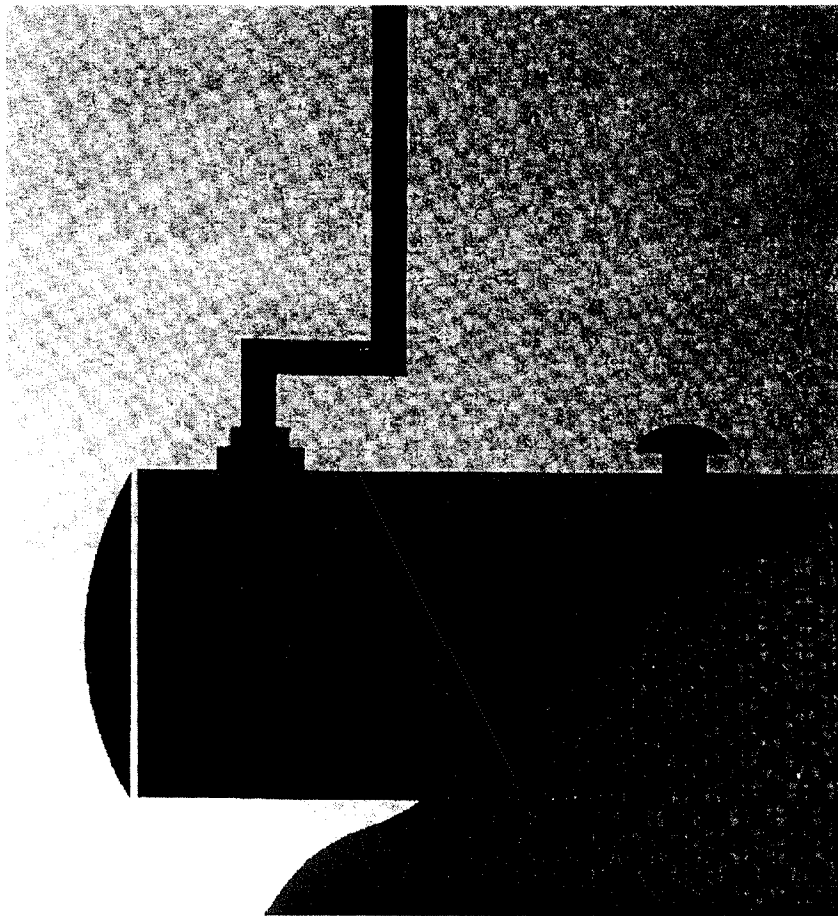
Each Pocket Colorimeter II Instrument comes complete as a ready-to-use kit that includes a reagent set (pre-measured unit dose reagents), sturdy custom carrying case, and manual.

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Expedited Site Assessment Tools For Underground Storage Tank Sites

A Guide For Regulators



Expedited Site Assessment Tools For Underground Storage Tank Sites

A Guide For Regulators

United States Environmental Protection Agency
Office of Underground Storage Tanks, OSWER

March 1997

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Field Methods For The Analysis Of Petroleum Hydrocarbons

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Chapter VI

Field Methods For The Analysis Of Petroleum Hydrocarbons

Analysis of soil, soil-gas, and groundwater samples in the field is an essential element of expedited site assessments (ESAs). Field managers require field-generated data in order to complete a site assessment in a single mobilization. In recent years many field methods for petroleum hydrocarbon analysis have been developed and improved. These technological improvements can change the way site assessments are conducted by providing reliable data in the field that can then be used to select subsequent sampling locations.

Historically, the analysis of contaminated media during UST site assessments has been completed off-site in fixed laboratories that use certified analytical methods. While these methods provide a very high data quality level (DQL), their results may take days or weeks and their cost is relatively high. In addition, many studies have shown that samples can undergo significant degradation during the shipping and holding times before analysis.

The development and improvement of many field methods have allowed site assessments to be performed more rapidly and completely than is feasible with off-site analysis. By combining field methods of different DQLs, ESAs can improve the resolution of contaminant distribution and minimize analytical costs. Low DQL (*i.e.*, screening) methods can be used to provide a high density of data to determine source areas (*i.e.*, zones of non-aqueous-phase liquid [NAPL] contamination). Higher DQL methods can be used to identify low concentrations or specific chemicals of concern at select locations (*e.g.*, leading edge of contaminant plume). Data from higher DQL methods can also be used as part of a quality control check for the field analytical program.

Exhibit VI-1 is a summary table of the primary selection criteria for eight commonly available field methods applicable for the analysis of petroleum hydrocarbons. It is followed by a brief discussion of the DQL system used in this chapter. The majority of the chapter is dedicated to discussions of the eight field methods listed in Exhibit VI-1. Each method is summarized with a capabilities and limitations table. A brief description and discussion of emerging technologies (*i.e.*, new technologies that are subject to significant innovation in the immediate future) appears at the end of the chapter. In addition, Appendix B, at the end of the manual, provides the reader with a table of relevant U.S. EPA test methods for petroleum hydrocarbons.

The chapter is organized so that readers can use the summary table (Exhibit VI-1) for initial selection of the most appropriate methods for a specific situation. They can then make a final selection by referring to the discussions of the individual methods that follow. The simpler, lower DQL methods are presented first.

Exhibit VI-1
Summary Table Of Field Methods For Petroleum Hydrocarbon Analysis

Test Method	Media ¹			Analyte	Data Quality Level ²	Analysis Time	Cost Per Sample ³	Skill Level	Limitations
	S	SG	W						
Detector Tubes		✓	✓	>100 specific compounds	1A/1B	5 to 15 min	\$8 to \$27	Low	High degree of cross-reactivity
Fiber Optic Chemical Sensors		✓	✓	VOCs and SVOCs ≥ C ₆	1A/1B	3 to 5 min	<\$1 to \$10	Low	Does not measure specific constituents
Colorimetric Test Kits	✓		✓	Aromatic hydrocarbons	1A/1B	10 to 20 min	\$17 to \$42	Low-Medium	Colors may be difficult to distinguish
TOV Methods With FID/PID	✓	✓	✓	Total VOCs	1A/1B	1 to 30 min	<\$1 to \$10	Low-Medium	Does not measure specific constituents
Turbidimetric Test Kit	✓			TPH of mid-range hydrocarbons (e.g., diesel fuel)	1B	15 to 20 min (25 per hour) ⁴	\$10 to \$15	Low-Medium	Not useful for gasoline
Immunoassay Test kits	✓		✓	TEX/PAHs/TPH	1B	30 to 45 min (5 to 8 per hour) ⁴	\$20 to \$60	Medium	Cross-reactivity may affect interpretation
Portable Infrared Detectors	✓		✓	TPH of hydrocarbons C ₆ to C ₂₆	2	5 to 20 min	\$5 to \$30	Medium	VOCs are not accurately analyzed
Field GC	✓	✓	✓	Specific VOCs and SVOCs	2/3	10 to 60 min ⁵	\$20 to \$70	Medium-High	Requires a skilled technician

¹ Soil (S), Soil-Gas (SG), Water (W)

² Data quality levels are discussed in further detail in the following text

³ Includes estimation of capital costs and disposables -- excludes labor

⁴ When run in batches

⁵ Longer times result when high quality method preparations are performed

Data Quality Levels

Data quality levels (DQLs) represent a classification system of analytical methods by the quality of data they provide. DQLs are one of several criteria that can be used for selecting an appropriate analytical method. Exhibit VI-2 presents the summary table of the DQL classification system used in this manual, which was adapted from the classification system developed by the New Jersey Department of Environmental Protection (1994). The levels are organized in a data quality hierarchy in which DQL 1 provides screening information, DQL 2 provides quantitative data, and DQL 3 provides the most rigorous quantitative data. Every state will have its own definition and requirements for various field analytical methods and its own DQLs, so a complete list of QA/QC procedures for each level is not provided.

Data Quality Level 1: Screening

DQL 1 screening methods are divided into two subgroups: 1A and 1B. Both are used for an initial screening of samples or for health and safety evaluations. DQL 1A provides a general indication of the presence of contaminants, DQL 1B provides relative numerical values. All DQL 1 methods:

- May require confirmation with higher DQL methods; and
- Detect the presence of classes or groups of constituents.

**Exhibit VI-2
Summary Of Data Quality Levels**

Data Quality Level	General Field Applications
1A: Qualitative Screening	General presence of contamination (e.g., "Yes/no," low/medium/high); health and safety
1B: Semiquantitative Screening	Approximation of contaminated zone; provides order of magnitude estimations (e.g., 10s, 100s, 1000s)
2: Quantitative Delineation	Delineation of specific contaminants
3: Quantitative Clean Zone	Regulatory monitoring, determining clean samples

Data Quality Level 1A: Qualitative Screening

DQL 1A is designated for initial screening of soil, soil gas, and groundwater by providing a "yes/no" indication of contamination. Measurements made with these methods may not always be consistent because of the lack of sample control and inherent method variability. As a result, clean samples cannot be determined from this level. Examples of DQL 1A methods include ambient air analysis or jar headspace using flame-ionization detectors (FIDs) and photoionization detectors (PIDs).

Data Quality Level 1B: Semiquantitative Screening

DQL 1B provides a rough, order of magnitude (*e.g.*, 10s, 100s, 1000s) estimate of contamination. It can be used for defining the location of known types of contamination. QA/QC procedures include a calibration curve generated using matrix spiked standards, regular calibration checks, and field blank/background samples. An example of DQL 1B is the data from some immunoassay test kit methods.

Data Quality Level 2: Quantitative--Delineation

DQL 2 methods provide reliable data for the delineation of contaminants during a site assessment. Typically, they are laboratory methods adapted for the field (*e.g.*, portable GC methods). DQL 2 methods:

- Measure individual constituents (*e.g.*, benzene) or groups of constituents (*e.g.*, BTEX, gasoline/diesel range organics);
- Produce data that are highly reproducible and accurate when appropriate QA/QC procedures are used; and
- Accomplish contaminant delineation, which may be correlated with a higher DQL method.

Data Quality Level 3: Quantitative--Clean Zone

DQL 3 methods are approved laboratory methods (*e.g.*, U.S. EPA SW-846 Laboratory Methods) and are intended to provide the most reliable data practicable. These methods can be used for confirming "clean" samples and for

regulatory monitoring. DQL 3 can be performed both off-site in a fixed laboratory or on-site in a mobile laboratory.

manufacturer (*e.g.*, p-xylenes, isopropanol). Calibrations must be checked daily or periodically between samples.

To measure hydrocarbon concentrations in vapor wells, a humidity tube is used to zero the probe to the humidity in which the measurement will be made. The probe sensor is then lowered to the desired depth. When the readings have stabilized, the measurement can be recorded. The time required to reach a stable reading is related to the temperature difference between the temperature at which the probe sensor was zeroed and the temperature of the well. Well-specific response factors may be used to obtain a specific concentration for that well. After the measurement is completed, the probe must remain above ground for 5 minutes to allow any vapors in it to dissipate.

Method Capabilities And Practical Considerations

FOCS are capable of detecting VOCs and SVOCs with six or more carbon atoms. Thus, benzene (C_6) can be detected while methane (C_1) cannot. The response of the sensor probe is directly related to the quantity of hydrocarbons present in a sample, calibrated to a p-xylene response. However, highly soluble constituents yield a lower response than less soluble constituents. For example, benzene, which is approximately 10 times more soluble in water than p-xylene, responds with one-tenth the sensitivity of p-xylene. In addition, the response is affected by temperature. FOCS, therefore, almost always require temperature compensation, which is usually built into the sensor. The optimal temperature range of FOCS is generally between 50° and 86° F (10° and 30° C).

Because the readings provide a relative value, a response factor (empirically determined by the manufacturer) must be used to estimate contaminant levels once the constituents and their relative ratios have been determined. The strongest correlation of results with GC analysis comes either from a single well monitored over time or from wells contaminated by the same source. Exhibit VI-6 presents a summary of FOCS method capabilities and limitations.

Colorimetric Test Kits

Colorimetric test kits provide qualitative or semiquantitative screening of aromatic hydrocarbons in soil and water. They can generally provide information about compound groups (*e.g.*, BTEX, PAHs) but can also help determine concentrations of specific compounds. A portable spectrophotometer has recently been developed to aid in the evaluation of concentrations in samples, however, the

Exhibit VI-6
Summary Of FOCS Method Capabilities
And Practical Considerations

	Water	Vapor
Compounds Detected	VOCs and SVOCs \geq C ₆ (Benzene)	
Lower Detection Limits	0.1 to 5 ppm	3 to 65 ppm
Limitations	<p>Does not measure specific constituents</p> <p>Concentrations at specific locations must be calculated by comparing historical DQL 3 results with FOCS results</p> <p>Free product saturates coating and exceeds meter scale</p> <p>Optimal temperature range is between 50° and 86° F.</p>	
Time For Analysis	3 to 5 minutes	
Difficulty of Procedure	Low	
Data Quality Level	1A/1B	
Cost Per Sample¹	<\$1 to \$10	

¹ Reflects the averaged cost over an extended period of time including consumables (e.g., calibration standards) and the capital cost of equipment, ranging from \$5000 to \$6900.

primary method of evaluation is by visual comparison of sample results with calibrated photographs of specific substances (e.g., gasoline, diesel fuel).

Operating Principles

Colorimetric test kits that are designed for hydrocarbon analysis create intensely colored aromatic compounds through the Friedel-Crafts alkylation reaction. This reaction utilizes a catalyst (e.g., AlCl₃) to attach an alkyl group to an aromatic hydrocarbon (e.g., benzene). In these test kits, an alkylhalide (e.g., carbon tetrachloride [CCl₄]) is typically used as both an extracting agent for the hydrocarbons and as a reagent. Once the catalyst is added, the reaction proceeds. The resulting color (e.g., orange, violet) provides information about the type of

constituent; the intensity of the color is directly proportional (within a specific range) to the concentration. Exhibit VI-7 presents a common Friedel-Crafts alkylation reaction utilized in colorimetric test kits.

Method Descriptions

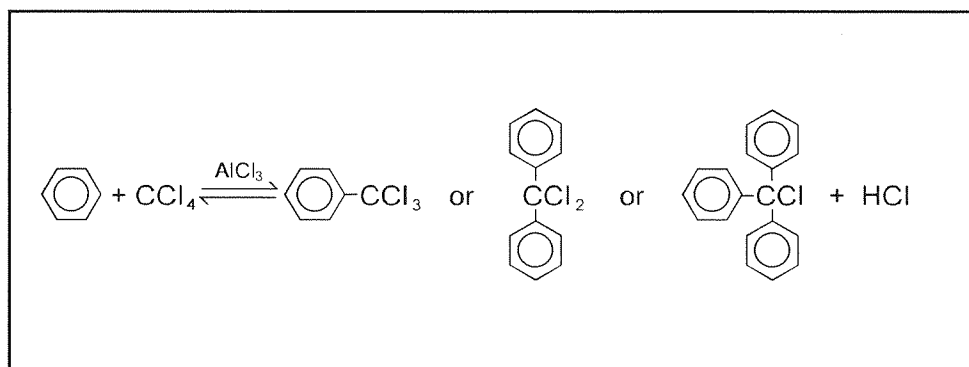
Colorimetric test kits are available for soil and water analysis. The kits provide the reagents and equipment needed for the extraction and colorimetric analysis of aromatic hydrocarbons. Color charts, created from known concentrations of various constituents, are used for comparison with field results to determine the constituents and their approximate concentrations.

Water Test Kit

The water test kit requires the following steps:

- Pour the water sample into a separatory funnel;
- Add the solvent/extract (an alkylhalide) to the sample, agitate it, and wait until solvent/extract has settled to the bottom of the separatory funnel;
- Drain the extract into a test tube;
- Add the catalyst and agitate it while the reaction proceeds between the aromatics and the alkylhalide; and
- Compare the color of the sample in the test tube (precipitate) with the color chart standard.

Exhibit VI-7 Example Of Friedel-Crafts Alkylation Reaction Utilized In Colorimetric Test Kits



Soil Test Kit

The soil test kit requires the following steps:

- Measure a soil sample;
- Add the solvent/extract to the soil, agitate it vigorously, and wait for the solvent/extract to separate;
- Pour the extract into a test tube;
- Add the catalyst and agitate; and
- Compare the color of the sample in the test tube (precipitate) with the color chart standard.

Method Capabilities And Practical Considerations

Colorimetric test kits can be used to analyze aromatic hydrocarbons (with particular sensitivity to PAHs) in soil and water. In soil, the detection limit is generally in the 1 to 10 ppm range; in water it is less than 1 ppm. Colorimetric test kits are effective for analysis of gasoline, diesel fuel, and other fuel oil contamination. A particular advantage of this method is that it is not dependent on analyte volatility, making it especially useful for older spills and for heavier fuel oils.

One of the major limitations of the method is that when comparing samples with the color chart photos, constituent concentrations and colors (*i.e.*, type of constituent) can be difficult to determine when constituent concentrations are low. In addition, if the contamination is a mixture of constituents, lighter aromatics (*e.g.*, BTEX) which turn to shades of orange will be hidden by heavier constituents (*e.g.*, PAHs) which turn to shades of violet. As a result, constituents present in the sample should be known before analysis.

There are a number of potential interferences for this type of analysis. First, the presence of chlorinated solvents may result in false positive analysis with water or soil. Second, color interferences for organic-rich or clayey soils may make color interpretation difficult. Clay soils may also pose additional problems because the sample tends to clump, making contaminant extraction difficult. Finally, the reaction products are sensitive to UV radiation, becoming darker with time and causing the potential for overestimation of constituent levels. Constituents and concentrations should, therefore, be determined within 30 minutes of color formation.

A health and safety issue involved with the use of this method is that analysis of all water samples and soil samples with hydrocarbon concentrations less than 1000 ppm requires a heptane-carbon tetrachloride solution to be used for sample extraction. Therefore, reagents and waste products must be properly handled and disposed of after use, typically, they are shipped back to the manufacturer. For analysis of soil samples with greater than 1000 ppm hydrocarbons, a much more environmentally safe heptane solution (without tetrachloride) can be used for extraction.

Both soil and water test kits provide data for screening level analysis. Because the soil test provides variable response to a wide range of aromatic hydrocarbons, has several interferences, and can be difficult to use, it is classified as a DQL 1A analysis. The water analysis is more accurate and allows for an order of magnitude determination of contamination. As a result, it is capable of providing DQL 1B analysis. A summary of the capabilities and practical considerations for analysis using colorimetric methods is shown in Exhibit VI-8.

Analysis With Reflectance Spectrophotometer

A portable reflectance spectrophotometer and associated software have been developed that allow objective measurement of color intensity. Future innovations may allow quantification of specific constituents and increase the upper level of measurement. It is available for approximately \$4,500.

Total Organic Vapor Analytical Methods With Flame Ionization And Photoionization Detectors

Total organic vapor (TOV) analytical methods detect the total volatile organic compounds in a sample. Although, they provide information about the relative magnitude of contamination, TOV methods are unable to distinguish specific compounds.

Operating Principles

There are two types of instruments commonly used in TOV analysis-- flame ionization detectors (FIDs) and photoionization detectors (PIDs).

Exhibit VI-8
Summary Of Colorimetric Test Kit Method Capabilities
And Practical Considerations

	Soil Test Kit	Water Test Kit
Compounds Detected	Monoaromatic and polyaromatic hydrocarbons	
Measuring Range	Benzene 1 to 200 ppm	Benzene 0.2 to 10 ppm
	Toluene 0.5 to 250 ppm	Toluene 0.2 to 10 ppm
	Gasoline 1 to 1,000 ppm	Gasoline 0.5 to 20 ppm
	Diesel 1 to 1,000 ppm	Diesel 0.5 to 20 ppm
	JP-5 1 to 2,000 ppm	Naphthalene 0.1 to 2.5 ppm
Limitations	<p>Mixtures of constituents may make colors difficult to distinguish without spectrophotometer.</p> <p>Investigators should know constituents present before analyzing samples</p> <p>UV light degrades the color of samples (<i>i.e.</i>, they become darker) approximately 30 minutes after color formation.</p> <p>Extraction of constituents may be difficult in clays.</p> <p>Organic and clay-rich soils may interfere with color.</p> <p>Carbon tetrachloride must be used, and properly disposed of, for analysis < 1000 ppm.</p>	
Time For Analysis	10 to 20 minutes	10 to 15 minutes
Difficulty Of Procedure	medium	low-medium
Data Quality Level	1A	1B
Cost Per Sample¹	\$17 to \$42	

¹ Initial 30 analyses cost \$42; subsequent analyses may cost as little as \$17.